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Indian Standard

METHYLENE CHLORIDE FOR DEGREASING PRIOR TO ELECTROPLATING — SPECIFICATION

UDC 661·723·21 : 621·357·7

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Electroplating Chemicals Sectional Committee, CHD 05

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

Methylene chloride is amongst the least toxic and hazardous of chlorinated solvents and is widely used as an industrial solvent, particularly, in metal degreasing operations.

A prime requirement for any vapour degreasing solvent is its ability to dissolve all greases, fats, oils, waxes, resins, gums, etc, that are generally used in metal working operations. The solvent cleaning power is the greatest for methylene chloride.

Vapour degreasing is a simple and effective process. When hot solvent vapours come into contact with a cooler object, they condense into liquid solvent on its surface, thereby, warming the object and dissolving the greasy matter. This solution is then drained off. When the surface temperature of the object reaches that of the vapour, condensation ceases and the clean, dry object can be removed from the degreaser.

The solvent shall also clean the object without corroding, oxidizing, staining, etching or fritting it and shall not reduce the gloss on the object.

Methylene chloride has low boiling point and is, therefore, also well suited for cleaning thermal switches, thermometers and other similar temperature sensitive parts. It also has the highest solvent power and can remove tough paint residues and hard-to-dissolve resins effectively. The energy efficiency is also the highest with methylene chloride.

Tough least toxic of the chloromethanes, methylene chloride is strongly narcotic and may cause dizziness and slight nausea. It is very dangerous to the eyes. It can, however, be handled safely, if sufficient precautions are taken (*see* also Annex A).

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance, with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value, should be the same as that of the specified value in this standard.

Indian Standard

METHYLENE CHLORIDE FOR DEGREASING PRIOR TO ELECTROPLATING — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and test for methylene chloride (dichloromethane) suitable for degreasing purposes.

2 REFERENCES

The Indian Standards given below are the necessary adjuncts to this standard:

IS No.	*Title*
74 : 1979	Methods of sampling and test for drying oil for paints (*second revision*)
197 : 1969	Methods of sampling and test for varnishes and lacquers (*first revision*)
878 : 1975	Graduated measuring cylinders (*first revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods : Part 1 Dangerous goods (*first revision*)
1448 [P : 11] : 1970	Methods of test for petroleum and its products [P : 11] : Freezing point of aviation fuels (*second revision*)
1448 [P : 24] : 1971	Methods of test for petroleum and its products [P : 24] : Kauri-Butanol value (*first revision*)
2362 : 1973	Determination of water by the Karl Fischer Method (*first revision*)
2837 (Part 2) : 1977	Porcelain crucibles and basins: Part 2 Basins (*first revision*)
4905 : 1968	Method of random sampling
5298 : 1983	Method for determination of distillation range and of distillation yield (*first revision*)

3 REQUIREMENTS

3.1 Description

Methylene chloride shall be a colourless liquid free from matter in suspension and shall have a sweet penetrating ether-like odour. It is slightly soluble in water but completely soluble in alcohol and ether. It shall conform essentially to the formula CH_2Cl_2.

3.2 The material shall also comply with the requirements prescribed in Table 1.

4 PACKING AND MARKING

The material shall be supplied in sealed and fully air-tight galvanized iron containers or as agreed to between the purchaser and the manufacturer.

4.2 Marking

4.2.1 The containers shall be suitably marked in red letters as POISONOUS and shall be labelled as shown in Fig. 5 of IS 1260 (Part 1): 1973. The label shall also bear the following clear and legible instructions:

> 'STORE IN A COOL, WELL VENTILATED PLACE AWAY FROM FIRE AND FLAMES, USE WITH ADEQUATE VENTILATION. AVOID PROLONGED BREATHING OF VAPOURS.'

4.2.2 The containers shall also be marked with the following particulars:

a) Indication of the source of manufacture;
b) Name of the material;
c) Net mass;
d) Batch number; and
e) Date of manufacture.

5 SAMPLING

The method of sampling and criteria for conformity shall be in accordance with Annex F.

6 TEST METHODS

6.1 Tests shall be conducted as prescribed in the relevant specifications or annex of this specification as given in col. 5 and col. 4 of Table 1 respectively.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Methylene Chloride for Degreasing Prior to Electroplating

(*Clasue* 3.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Distillation range at a pressure of 1 bar	2 to 97 percent by volume between 39 to 40	—	5298 : 1983
ii)	Freezing point, °C	— 97	—	1448 [Part 11] : 1970
iii)	Relative density 27/27°C	1·310 to 1·320*	B	—
iv)	Refractive index 27°C	1·421	—	Clause 8 of 74 : 1979
v)	Viscosity 27°C (cps)	0·329	—	Clause 17 of 197 : 1969
vi)	Kauri-butanol value	136	—	1448 [P : 24] : 1971
vii)	Non-volatile matter, percent by mass, *Max*	0·01	C	—
viii)	Moisture, percent by mass, *Max*	0·05	—	2362 : 1973
ix)	Acidity (as HCl), percent by mass, *Max*	0·01	D	—
x)	Free chlorine	To pass test	E	—

*The correction factor within the range 15 to 35°C is +0·0016 for each degree celsius fall and —0·0016 for each degree celsius rise in temperature.

ANNEX A

(*Foreword*)

HAZARDS AND SAFETY PRECAUTIONS FOR METHYLENE CHLORIDE

A-1 Normally two types of hazards are foreseen vapour inhalation and decomposition.

A-2 The effect of inhaling solvent vapours at toxic levels can be acute or chronic or both. An acute, or one-time, high exposure can cause anesthetic effects, such as light headedness, dizziness, loss of consciousness, or, if the exposure involves an excessively high concentration of solvent vapours, it can even cause death. Chronic exposure, under which a person continually and repeatedly inhales concentrated solvent vapours, can lead to liver injury.

A-2.1 Workers can be protected from these potential hazards by observance of standards set by various health agencies.

A-3 Vapours of chlorinated solvents, even in relatively low concentrations, may decompose in contact with open flames or hot surfaces. In thermal decomposition, the chlorinated solvents breakdown to form hydrogen chloride, carbon dioxide, carbon monoxide and phosgene. Should this happen, it will require unprotected workers to leave the area quickly, before other hazards arise.

A-3.1 For safety, chlorinated solvent vapours should be vented in order to avoid heat sources. Any welding operations should be stopped immediately, if the odour of chlorine or hydrogen chloride is noticed.

ANNEX B

[*Clause* 3.2, *Table* 1, *Sl No.* (iii)]

DETERMINATION OF RELATIVE DENSITY

B-1 GENERAL

Relative density is determined using specific gravity bottle or pyknometer.

B-2 APPARATUS

B-2.1 Specific Gravity Bottle or Pyknometer

With well-fitting ground-glass joints. To calibrate, clean and dry the bottle or pyknometer thoroughly, weigh and then fill with water, recently boiled and cooled to below 27°C, after removing the cap of the side arm. Fill to overflowing by holding the bottle pyknometer on its side in such a manner as to prevent entrapment of air bubbles, insert the stopper and immerse in a water bath maintained at 27 ± 0·2°C. Keep the entire bulb completely covered with water and hold at that temperature for 30 minutes. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, replace the cap, cool to a lower temperature and weigh. Calculate the mass of water. The use of constant gravity bottle of about 50 ml capacity is recommended.

B-2.2 Water Bath — capable of maintaining temperature at 27 + 0·2°C.

B-2.3 Thermometer

Any convenient thermometer of a suitable range with 0·10 or 0·2°C sub-divisions. The thermometer should be checked against a standard thermometer that has already been calibrated.

B-3 PROCEDURE

Fill the bottle or pyknometer with the material previously kept at about 27°C to overflowing, holding the apparatus on its side in such a manner as to prevent entrapment of air bubbles after removing the cap of the side arm. Insert the stopper, immerse in the water bath maintained at 27 + 0·2°C, and hold for 30 minutes. Carefully wipe off any liquid which has come out through the capillary opening. Remove the apparatus from the bath, clean and dry it thoroughly. Replace the cap, cool to a lower temperature and weigh.

B-4 CALCULATION

$$\text{Relative density at } 27/27^\circ\text{C} = \frac{A - B}{C - B}$$

where

A = Mass of the specific gravity bottle or pyknometer filled with the material at 27°C.

B = Mass of the specific gravity bottle of pyknometer, and

C = Mass of the specific gravity bottle or pyknometer filled with water at 27°C.

ANNEX C

[*Clause* 3.2, *Table* 1, *Sl No.* (viii)]

DETERMINATION OF RESIDUE ON EVAPORATION

C-1 APPARATUS

C-1.1 Porcelain Basin — 150 ml capacity [*see* IS 2837 (Part 2) : 1977].

C-1.2 Water-Bath

C-1.3 Oven — capable of maintaining temperature between 105°C to 110°C.

C-2 PROCEDURE

Clean the basin and dry in a desiccator. Weigh it to the nearest 0·000 2 g. Take 100 ml of the material in the basin and weigh again to the nearest 0·01 g. Evaporate the material over a water-bath in a fume cupboard to practically dryness. Further heat in the oven at 105 to 110°C for 30 minutes. Cool in a desiccator and weigh accurately. Repeat the heating for 15 minutes, followed by cooling and weighing till two consecutive weighings do not differ by more than 0·3 mg.

C-3 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{M_2 - M}{M_1 - M} \times 100$$

where

M_2 = mass in g of the basin with the dry residue,

M = mass in g of the basin, and

M_1 = mass in g of the basin with 100 ml of the material.

ANNEX D

[*Clause* 3.2, *Table* 1, *Sl No.* (ix)]

DETERMINATION OF ACIDITY

D-1 GENERAL

Acidity is determined by titrating a known quantity of material with a standard alkali.

D-2 REAGENTS

D-2.1 Phenolphthalein Indicator Solution

Prepare the indicator by dissolving 0·5 g of the phenolphthalein in 100 ml of ethanol (95 percent) and make it faintly pink by the addition of dilute sodium hydroxide solution.

D-2.2 Standard Sodium Hydroxide Solution — 0·01 N.

D-3 PROCEDURE

Place 100 ml of freshly boiled and cooled distilled water (neutral to phenolphthalein indicator solution) in a 300-ml glass-stoppered conical flask. Add 100 ml of the methylene chloride and shake vigorously. Allow the layers to separate, add 0·5 ml of the phenolphthalein indicator and titrate with 0·01 N sodium hydroxide using a micro-burette to the end point.

D-4 CALCULATION

$$\text{Acidity (as HCl), percent by mass} = 3{\cdot}65 \frac{V_1 N}{V D}$$

where

V_1 = volume in millilitres of standard sodium hydroxide solution,

N = normality of standard sodium hydroxide solution,

V = volume in millilitres of the material taken for the test, and

D = relative density of the material.

ANNEX E

[*Clause* 3.2, *Table* 1, *Sl No.* (x)]

TEST FOR FREE CHLORINE

E-1 OUTLINE OF THE METHOD

The material is shaken with a 3, 3′-dimethylnaphthidine solution and the colour developed, if any, is noted.

E-2 APPARATUS

E-2.1 Graduated Measuring Cylinder — 50 ml, glass stoppered (*see* IS 878 : 1975).

E-3 REAGENTS

E-3.1 3, 3′-Dimethylnaphthidine Solution

Dissolve 0·01 g of finely ground 3, 3′dimethylnaphthidine in 5 ml of glacial acetic acid and dilute rapidly with water to 200 ml. Store the solution in the dark.

E-4 PROCEDURE

To 50 ml of the material contained in the graduated measuring cylinder, add 5 ml of the 3, 3′-dimethylnaphthidine solution and shake the cylinder for 30 seconds.

E-4.1 The material shall be considered as showing no free chlorine if no pink colour is developed.

ANNEX F

(*Clause* 5)

SAMPLING OF METHYLENE CHLORIDE FOR ELECTROPLATING

F-1 GENERAL REQUIREMENTS OF SAMPLING

F-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

F-1.2 The sampling instrument shall be clean and dry.

F-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

F-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

F-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

F-1.6 The sample containers shall be of such a size that they are almost three-fourth filled by the sample.

F-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling, the date of sampling and details given under **3.2**.

F-2 SCALE OF SAMPLING

F-2.1 Lot

All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

F-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

F-2.3 The number of containers to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected from Lots of Different Sizes

(*Clause* F-2.3)

Lot Size *N* (1)	Sample Size *n* (2)
3 to 15	3
16 ,, 40	4
41 ,, 110	5
111 ,, 180	6
181 ,, 300	7
301 ,, 500	8
501 ,, 800	9
801 and above	10

F-2.3.1 These containers shall be selected at random. In order to ensure the randomness of selection, random number tables may be used (*see* IS 4905 : 1968). In case the random number tables are not available, the following procedure may be used.

> Arrange all the containers in the lot in a systematic manner and starting from any one, count them as 1, 2,.........up to r where r is the integral part of N/n; N and n being the lot size and sample size, respectively. Every rth container thus counted shall be withdrawn to constitute the sample.

F-3 INDIVIDUAL SAMPLES AND COMPOSITE SAMPLE

F-3.1 From each of the containers selected according to **F-2.3**, a representative portion of the material, about 300 ml in volume, shall be drawn. From each of these individual portions, an equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample not less than 1 000 ml in volume. The composite sample shall be transferred to clean bottles and labelled with full identification particulars of the sample.

F-4 NUMBER OF TESTS

F-4.1 Tests for the determination of the characteristics given in Table 1 shall be carried out on the composite sample.

F-5 CRITERIA FOR CONFORMITY

F-5.1 For declaring the conformity of the lot to the requirements of all the characteristics, the test results on the composite sample shall, meet the corresponding requirement specified.

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Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :	Telephone
Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	331 01 31 331 13 75
Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 84 99, 37 85 61, 37 86 26, 37 85 62
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